

**NASA TECHNICAL
MEMORANDUM**

NASA TM-73772

(NASA-TM-73772) THE EFFECT OF
MICROSTRUCTURE ON HYDROGEN EMBRITTLEMENT OF
THE NICKEL-BASE SUPERALLOY, UDIMET 700
(NASA) 32 P HC A03/MP A01

N78-22232

CSSL 11P

G3/27

Unclas
16618

NASA TM-73772



THE EFFECT OF MICROSTRUCTURE ON HYDROGEN EMBRITTLEMENT
OF THE NICKEL-BASE SUPERALLOY, UDIMET 700

by Hugh R. Gray

National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135

ABSTRACT

Material from a single heat of cast and wrought Udimet 700 was processed and/or heat treated to produce five material conditions with identical chemical compositions but with distinct microstructural variations, and then evaluated for susceptibility to hydrogen embrittlement. Two prealloyed powder conditions exhibited significantly improved resistance to hydrogen embrittlement, as compared to wrought material. No degradation in notch or smooth tensile strengths occurred, and average ductilities of 25 percent reduction of area were determined for 2 hydrogen evaluation procedures. For the most severe hydrogenation procedure, ductility levels were reduced to 15 percent. These improvements were attributed to cleaner grain boundaries and decreased grain size.

SUMMARY

Material from a single heat of wrought Udimet 700 was processed and/or heat treated to produce five material conditions with identical chemical compositions but with distinct microstructural variations. Specifically, the alloy was evaluated in three wrought forms - standard heat treatment, modified heat treatment, and standard heat treatment plus an intermediate cold working step. The alloy was also evaluated in two extruded from atomized powder forms - standard heat treatment, and a "yo-yo" heat treatment. These materials were then subjected to baseline tensile (smooth and notched) testing in air or argon at 23° and 675° C and to three hydrogen evaluation procedures - tensile testing in 3.5 MN/m² hydrogen; exposure in 0.1 MN/m² hydrogen at 675° C for 100 hours followed by tensile testing in air; and exposure in 3.5 MN/m² hydrogen at 675° C for 100 hours at a creep stress of 690 MN/m², followed by tensile testing in air.

Both of the Udimet 700 powder conditions exhibited significantly improved resistance to hydrogen embrittlement. No degradation in notch or smooth tensile strengths occurred, and average ductilities of about 25 percent reduction of area were determined for 2 of the 3 hydrogen evaluation procedures studied. For the most severe hydrogenation procedure ductility levels were reduced from approximately 28 to 15 percent. This improved tolerance for hydrogen is attributed to

finer grain size and cleaner grain boundaries. In addition the susceptibility of wrought Udimet 700 to embrittlement could be improved by either cold working or by eliminating the carbide precipitation heat treatment step.

By proper consideration of such microstructural features, it is suggested that nickel-base superalloys with tailored microstructures may provide reliable service in hydrogen environments.

These test results provide further evidence that Hydrogen Environment Embrittlement and Internal Reversible Hydrogen Embrittlement occur by the same mechanism. They differ only with respect to the initial location and supply of hydrogen, and its rate of delivery to and extent of trapping at critical microstructural features.

INTRODUCTION

Previous investigations (refs. 1 and 2) have demonstrated that wrought Udimet 700 (registered Trademark, Special Metals Corporation) is extremely sensitive to hydrogen embrittlement, as are other nickel-base superalloys used for high temperature structural applications (refs. 3 to 5). For example, when tested in gaseous hydrogen at 31 to 52 MN/m² pressure, Udimet 700 exhibited a notch tensile strength at room temperature of 970 MN/m² and a reduction of area of 7 percent, compared with baseline values in air of 1470 MN/m² and 17 percent respectively, see figure 1. It is also evident from this figure that the alloy's susceptibility to hydrogen embrittlement extends over a very wide temperature range, 23° to 700° C.

Udimet 700 is also substantially embrittled at room temperature at a hydrogen pressure of only 3.5 MN/m² (ref. 2). Specifically, as shown in figure 2, the notch tensile strength of wrought Udimet 700 in two heat treated conditions is reduced from approximately 1600 MN/m² to 800 MN/m² when tested in hydrogen. Material heat treated in the standard Udimet 700 manner and that given both the standard and "yo-yo" (refers to sequence of heat treating steps, see table I) heat treatments were embrittled to similar levels. The effect of this combined heat treatment on Udimet 700 was evaluated because Astroloy, an alloy very similar to Udimet 700 but with a "yo-yo" heat treatment had been shown to be quite resistant to hydrogen embrittlement (refs. 2 and 3).

Predominately intergranular fracture modes have been observed previously for hydrogen embrittled Udimet 700 as well as other nickel-base superalloys. The objective of this current investigation was to determine the effect of altered grain size and distribution of grain boundary phases and impurities on the susceptibility of Udimet 700 to hydrogen embrittlement.

Material from a single heat of wrought Udimet 700 was processed and/or heat treated to produce five material conditions with identical chemical compositions but with distinct microstructural variations. These material conditions were in-

tended to be appropriate for real structural applications. Specifically, the alloy was evaluated in three cast plus wrought forms - standard heat treatment, modified heat treatment, and standard heat treatment plus an intermediate cold working step. The alloy was also evaluated in two extruded from atomized powder forms - standard heat treatment, and a "yo-yo" heat treatment.

These five material conditions were then subjected to baseline smooth and notched tensile tests in air or argon at 23° and 675° C and to three different hydrogen evaluation procedures - tensile testing in 3.5 MN/m² hydrogen gas; exposure in 0.1 MN/m² hydrogen at 675° C for 100 hours, followed by tensile testing in air; and exposure in 3.5 MN/m² hydrogen at 675° C for 100 hours with an applied creep stress of 690 MN/m², followed by tensile testing in air. These three hydrogenation procedures were selected to span a spectrum of potential service applications. Relative susceptibility of these five material conditions to embrittlement by hydrogen was correlated with various microstructural features by conventional light, scanning and replica electron microscopy.

MATERIALS, SPECIMENS, AND PROCEDURES

Materials

The Udimet 700 material conditions, heat treatments, and resultant microstructural features evaluated in this investigation are listed in table I.

Material conditions 1, 2, and 3. - This 1.9-cm-diameter hot-rolled bar stock, from the same heat used in a previous investigation (ref. 2), was received in a partially heat treated condition (1175° C/4 hr + 1080° C/4 hr). It was then given the two remaining aging treatments, as per standard wrought Udimet 700 procedures (ref. 6). This material will hereafter be referred to as condition 1. An additional lot of this as-received bar stock was given only the 760° C aging treatment (condition 2). A third lot of as-received stock was swaged at room temperature in several successive reductions to achieve a total of 50 percent reduction of area, followed by the remaining two standard aging treatments (condition 3).

Material conditions 4 and 5. - Additional as-received bar stock from the same heat was vacuum remelted by a commercial vendor, argon atomized, separated into +100 and -100 mesh lots, and stored under argon. The -100 mesh (<150 μ m diameter) powder was canned under vacuum in type 304 stainless steel cans (16 cm in length and 7.6 cm in diameter). The electron-beam welded cans were preheated for 1 hour at 815° C and then heated for 1.5 hours at the extrusion temperature of 1120° C. The cans were extruded at a reduction ratio of 16 to 1 through a die having a 90° included angle to a final diameter of 1.9 centimeters, with the diameter of the Udimet 700 core approximately 1.1 centimeters. Some of these extruded bars were

then heat treated according to the standard Udimet 700 procedure described above and some to a "yo-yo" procedure (ref. 7), see table I. These materials will hereafter be referred to as conditions 4 and 5, respectively.

Compositions. - The chemical compositions of both the wrought bar stock and the extruded powder product were determined by arc emission spectroscopy (ref. 8). These results are given in table II, together with the nominal specification range (ref. 6) and vendor certified analysis of Udimet 700. The composition of the wrought bar stock agrees well with that reported by the vendor, with the unexplained exception of Al and Mo, both of which were determined to be greater than the vendor's results and with Al greater than the specification maximum.

Of more significance to this investigation, however, is the excellent agreement of analyses between the wrought (conditions 1, 2, and 3) and extruded powder (conditions 4 and 5) materials. No significant changes in major alloying elements resulted during atomization or consolidation. While the nitrogen and oxygen levels of the extruded powder product increased by approximately 20 and 75 ppm respectively, the resulting levels are typical of high quality atomization procedures.

Specimens

The smooth and notched specimens used in this investigation are shown in figures 3(a) and (b), respectively. The full size specimens were used for material conditions 1 and 2, while the slightly smaller specimens (values shown parenthetically for threaded ends and gage diameters) were used for the smaller diameter bar stock of material conditions 3, 4, and 5. Specimens were machined from fully heat treated stock and were inspected by both X-ray and dye penetrant techniques.

Procedures

The three evaluation procedures used in this investigation are summarized in table III. The following sections of this report describe in detail these evaluation procedures.

Evaluation procedure A. - One series of both smooth and notched specimens was tensile tested at 23° C and 675° C at a crosshead speed of 0.13 cm/min in 3.5 MN/m² hydrogen gas by Pratt & Whitney Aircraft, West Palm Beach, Florida. A multi-step purge and pressurization procedure was used to assure oxygen contamination levels of less than 1 ppm (for details see refs. 2 and 3). Baseline tensile testing was performed at Lewis at 23° C and 675° C at a crosshead speed of 0.13 cm/min in ambient pressure air. The results of all of these tensile tests are listed table IV, together with post-test hydrogen analyses of selected specimens which will be discussed later in this report.

Evaluation procedure B. - Another series of both smooth and notched specimens was exposed in hydrogen at 0.1 MN/m^2 pressure at 675°C for 100 hours. After this exposure the tensile properties of the materials were determined at 23°C and 675°C in ambient pressure air at a crosshead speed of 0.13 cm/min . Baseline specimens were subjected to a similar thermal exposure in argon and then tensile tested as described above. All of these exposures and tensile tests were conducted at Lewis. Test results and hydrogen analyses are listed in table V.

Evaluation procedure C. - A final series of smooth specimens was creep-exposed in hydrogen at 3.5 MN/m^2 pressure at 675°C for 100-plus hours at an applied creep stress of 690 MN/m^2 (at PWA - Florida). This exposure resulted in approximately 0.2 percent creep strain. After this exposure tensile properties were determined at 23°C in air. Baseline specimens were creep-exposed in ambient pressure air at 675°C for 100 hours at a stress of 690 MN/m^2 . All exposures and tests except the hydrogen creep-exposures were conducted at Lewis and results are listed in table VI.

Hydrogen analyses. - In addition to the hydrogen analyses reported in table II (0.6 ppm for all as-received material conditions), additional hydrogen analyses were performed on selected specimens after each of the three types of hydrogen evaluation procedures, see tables IV to VI. These tests were standard vacuum fusion analyses of a small sample (0.2 g) cut from adjacent to the fracture surface of tested specimens.

Metallography. - Light microscopy and replica electron microscopy were used to examine all material conditions. Scanning electron microscopy was used to examine selected fracture morphologies.

RESULTS AND DISCUSSION

Tensile Properties

Notch tensile strength. - As discussed in the INTRODUCTION, tensile testing of sharply notched specimens has been a commonly used method of determining susceptibility of alloys to embrittlement in gaseous hydrogen. The notch tensile strengths of all five Udimet 700 material conditions were determined at room temperature and are shown in figure 4. The results of figure 4(a) were determined in 3.5 MN/m^2 hydrogen (evaluation procedure A, table IV), while those in figure 4(b) were determined on specimens exposed in hydrogen at 675°C and 0.1 MN/m^2 pressure for 100 hours (evaluation procedure B, table V).

Several important observations can be made from these test results. First, Udimet 700 in its standard wrought and heat treated form (material condition 1) is

severely embrittled when tested in 3.5 MN/m^2 gaseous hydrogen (evaluation procedure A). Specifically, this material has a tensile strength of 1600 MN/m^2 in air and only 1170 MN/m^2 in hydrogen. These results are comparable to those determined in previous investigations of this material, see both figures 1 and 2.

Second, this same material condition did not show any degradation of notch tensile strength when tested after exposure in hydrogen at 675°C and 0.1 MN/m^2 pressure for 100 hours (evaluation procedure B), even though this exposure resulted in a twenty-fold increase in the analyzed hydrogen content (0.6 to 12 ppm) of the material, as will be discussed later in this report.

Third, all four of the other material conditions in which Udimet 700 was tested exhibited substantially greater resistance to embrittlement when tested in hydrogen (evaluation procedure A) than did the alloy in its standard condition. As shown in figure 4(a), the two powder conditions (4 and 5) of the alloy appeared quite resistant to embrittlement, while the material with the modified heat treatment and the cold-worked material (conditions 2 and 3) exhibited slight reductions in notch tensile strength of 1 and 3 percent, respectively. Similarly, after exposure in hydrogen (evaluation procedure B), the powder conditions and the modified heat-treatment condition exhibited degradation of less than 2 percent, while the cold-worked material suffered a loss of about 10 percent in notch tensile strength, figure 4(b).

It is apparent from the data in figure 4 that, with the exception of standard Udimet 700 tested in hydrogen, the other four material conditions investigated exhibited relatively small amounts of degradation in notch tensile strength at 23°C . Although not shown in any figure, none of the five material conditions exhibited reductions of notch tensile strength at 675°C of more than 3 percent, for either evaluation procedure A or B (for substantiating test data, see tables IV and V). Hence, notch tensile strength results are not considered a sensitive enough criterion for determining the role of microstructural variables on sensitivity to hydrogen embrittlement.

Tensile reduction of area. - In an effort to discriminate the influence of hydrogen on these various material conditions, the reduction of area values determined on smooth tensile specimens were evaluated and those determined at 23°C are shown in figure 5. It is evident from these data that reduction of area is indeed a sensitive criterion for determining the degree of hydrogen embrittlement among the five material conditions. For example, as shown in figure 5(a), for evaluation procedure A (testing in 3.5 MN/m^2 hydrogen) reduction of areas ranged from 7 percent for material condition 1 to 26 percent for material condition 5. These levels represent losses of about 70 to 25 percent from baseline (air) ductility levels, respectively. Only the most severely embrittled material condition 1 exhibited a significant decrease in ultimate tensile strength (from 1400 to 1055 MN/m^2 , see table IV).

Although not shown in a figure, tensile testing in hydrogen at 675° C indicated losses in ductility of 40 to 50 percent for material conditions 1, 2, and 3, whereas material condition 4 suffered a loss of only 10 percent, and condition 5 was not embrittled at all (see data in table IV).

Similar effects were determined for these material conditions after exposure in hydrogen at 675° C and 0.1 MN/m² pressure for 100 hours (evaluation procedure B), figure 5(b). Specifically, with the exception of the cold-worked material (condition 3), ductility levels ranged from 16 percent for material condition 1 to 28 percent for material condition 5. These values represent losses of about 30 to 3 percent from baseline (argon exposure) ductility levels, respectively. The cold worked material (condition 3) had relatively good resistance to embrittlement (8 percent R. of A.), although its baseline ductility level was quite low (9 percent R. of A.). None of the material conditions tested suffered any significant loss of ultimate tensile strength at 23° C, or tensile strength and ductility at 675° C.

All five material conditions exhibited less severe embrittlement when tested in air after this exposure (evaluation procedure B) than they did when tensile tested directly in gaseous hydrogen (evaluation procedure A). These results demonstrate that such a hydrogen exposure is less deleterious to Udimet 700 than testing directly in hydrogen.

However, when the hydrogen exposure at 675° C for 100 hours is conducted at a gas pressure of 3.5 MN/m² and a creep stress of 690 MN/m², all five material conditions tested exhibited severe embrittlement, see figure 5(c). Specifically, reductions of area values ranged from 6 to 15 percent, which for all five material conditions represent losses of about 40 to 60 percent from baseline (air) ductility levels. In addition, material conditions 1, 2, and 4 also suffered losses in ultimate tensile strength at 23° C ranging from 5 to 10 percent (see table Va). Material conditions 3 and 5 did not suffer significant losses in tensile strength.

Based on the above discussion of mechanical property results, one can conclude that: the most severe hydrogenation evaluation procedure is the high pressure (3.5 MN/m²) hydrogen creep-exposure at 675° C (C); the evaluation procedure with intermediate severity consists of tensile testing directly in high pressure hydrogen at 23° C (A); and the least severe hydrogenation evaluation procedure is the ambient pressure (0.1 MN/m²) hydrogen exposure at 675° C (B).

Based on an overall evaluation, see table VII, of tensile strength and ductility results at both 23° and 675° C for all three hydrogenation conditions, Udimet 700 in material condition 5 exhibited the greatest resistance to embrittlement by hydrogen. The overall ranking of the other Udimet 700 material conditions, although not as clear cut as that of material condition 5, has been judged as follows: material condition 4, condition 2, condition 3, and finally, the most susceptible,

the standard Udimet 700 material condition 1. The relationship between these rankings and the alloy microstructures will be discussed later in this report.

Hydrogen Analyses

Vacuum fusion chemical analyses were performed on selected test specimens after baseline exposures and after each of the three hydrogen evaluation procedures. A total of nine analyses were made of baseline (air or argon exposures) specimens of both wrought (material condition 1) and powder (material conditions 4 and 5) specimens. The average hydrogen content of these baseline analyses was 0.6 parts per million by weight (ppm) with specific analyses ranging from 0.5 to 0.8 ppm, see tables IV and V.

Tensile testing directly in high pressure hydrogen (evaluation procedure A) did not result in any detectable increase in the hydrogen content of the test specimens. Specifically, four analyses ranged from 0.2 to 0.5 ppm which, although slightly below baseline values, should be considered equivalent to baseline analyses within the range of experimental accuracy of this analysis technique. It should be noted that these analyses were conducted on small (0.2 g) samples cut from specimen gage sections adjacent to fracture surfaces. Hence, these analyses represent the average hydrogen content of the entire sample. It is likely that local hydrogen concentrations at and immediately below the specimen gage and fracture surfaces are substantially greater than these bulk values, as has been demonstrated with an ion microanalyzer in a previous investigation (ref. 9).

Exposure in 0.1 MN/m^2 hydrogen at 675°C for 100 hours (evaluation procedure B) resulted in 20-fold increases in hydrogen contents with 12 ppm hydrogen detected in each of the four different material conditions analyzed (see table V).

Exposure in 3.5 MN/m^2 hydrogen at 675°C for 100 hours at creep stress of 690 MN/m^2 (evaluation procedure C) resulted in 100-fold increases (to 60 ppm) in hydrogen contents for all the material conditions analyzed (see table VI). Both of these analyzed hydrogen levels are in good agreement with calculated, uniform concentration based on permeability data for 100 hours of exposure at 675°C .

Hence, it is apparent from these analyses that: (1) for a given hydrogenation condition, all material conditions absorbed equivalent amounts of hydrogen and, therefore, relative susceptibility to embrittlement can not be attributed to varying absorption rates, and (2) the most severe evaluation procedure (C) resulted in a 100-fold increase in specimen hydrogen content, the least severe evaluation procedure (B) resulted in a 20-fold increase, while the intermediate severity evaluation procedure (A) did not result in any detectable increase in bulk hydrogen content.

Microstructures and Fractography

Udimet 700 condition 1. - The microstructure of the standard wrought Udimet 700 stock used in the investigation (identical to that of ref. 2) is shown in figure 6. This material condition consists of large, equiaxed grains with numerous annealing twins and large intragranular carbide particles. The average grain size is ASTM 2 (range 0 to 4). The grain boundaries are lined with an almost continuous network of carbides, borides and massive gamma prime particles. The observable matrix gamma prime is present in a bimodal distribution with average diameters of 0.08 and 0.35 μm . All of these microstructural features are typical of those expected to be found in Udimet 700 alloy subjected to the standard heat treatment (refs. 1 and 2) and are summarized in table I.

Scanning electron fractographs of specimens after tensile testing at 23 $^{\circ}$ C in air, in hydrogen (evaluation procedure A), and after hydrogen exposure (evaluation procedure C) are shown in figures 7(a) to (c), respectively. It is immediately evident from these figures that Udimet 700 in material condition 1 fractures in a predominately intergranular mode when tested in baseline air (fig. 7(a)), or after exposure in hydrogen (fig. 7(c)), and a totally intergranular mode when tested in hydrogen (fig. 7(b)). Subsurface intergranular cracking occurred to approximately the same extent in not only both of the hydrogenated specimens but also in the baseline specimen tested in air. The grain boundary structure that is evident at higher magnification also appears similar for all three conditions shown in figure 7.

In summary other than a slight change from predominately to totally intergranular fracture, no detectable differences were observed between specimens tested under baseline conditions and hydrogenated conditions. In addition, specimens tested in hydrogen and those exposed in hydrogen did not exhibit any significant fractographic variations. This latter observation is important from a hydrogen embrittlement mechanistic viewpoint, as will be discussed in the CONCLUDING REMARKS section of this report.

Udimet 700 condition 2. - As anticipated from the deliberate omission of the heat treating step at 845 $^{\circ}$ C, the grain boundaries in Udimet 700 in material condition 2 are substantially cleaner than in condition 1, see figure 8. Specifically, the almost continuous network of grain boundary carbides, borides and gamma prime precipitates found in material condition 1 was virtually eliminated with the modified heat treatment given material condition 2. Only a few isolated, but massive, grain boundary phases were found in this microstructure. All other microstructural features, such as grain size, intragranular carbides, and bimodal gamma prime precipitate sizes appeared to be unchanged from those determined previously and discussed above for material condition 1.

As evident from figure 9, these cleaner grain boundaries accompanied a change in the fracture mode of this material to mixed intergranular and transgranular. Once again, as far as could be determined, testing in or exposure to hydrogen did not alter any features of the fracture mode compared with testing in air.

Udimet 700 condition 3. - The effects of cold working the alloy 50 percent before giving it the final two standard aging heat treatments at 845° and 760° C are evident from the micrographs shown in figure 10. The initially equiaxed grains have been slightly elongated in the direction of working. Large intragranular carbides are still present and additional small ones have precipitated along the numerous deformation bands created within the grains by the working process. At the highest magnification it is apparent that the working process has deformed the large matrix gamma prime particles which had precipitated during the two heat treating steps prior to working. These deformed gamma prime precipitates, as well as the increased density of dislocations, could result in more trapping of hydrogen, as will be discussed in a subsequent section of this report. An almost continuous network of grain boundary gamma prime, carbides and borides are still evident in this material, in spite of the goal of "cleaning up" the grain boundaries by promoting substantial intragranular carbide precipitation along the deformation bands.

As evident from the scanning electron fractographs presented in figure 11, cold worked Udimet 700 fractures in a substantially different mode than did the standard Udimet 700 in condition 1 (compare fig. 11 with fig. 7). The fracture mode has changed from intergranular to primarily transgranular with only a few indications of intergranular fracture. There were no detectable differences between specimens tested in air (fig. 11(a)) or in hydrogen (fig. 11(b)). Both specimens exhibited complete shear lips, although reduction of area values were only 12 and 9 percent, respectively.

Udimet 700 condition 4. - The overall grain size and morphology of powder metallurgy Udimet 700 is most easily discernable when examined in the solution annealed condition, as shown in figure 12(a). This average grain size, ASTM number 10, is typical of a recrystallized powder metallurgy product after extrusion at 1120° C and solution annealing at 1175° C. This annealing temperature is slightly above the gamma prime solvus temperature of 1160° C for this material (determined experimentally by differential thermal analysis).

The microstructure of the fully heat treated material is shown in figure 12(b) at intermediate magnification and in figure 12(c) at high magnification. It is immediately evident that although this material has some large gamma prime precipitates at the grain boundaries, it does not have either the large intragranular carbides or the continuous network of grain boundary carbide, boride and gamma prime phases that the wrought Udimet 700 had in the standard condition 1. In fact only a few small carbides are visible in the microstructure indicating that the

atomization and powder metallurgy process resulted in a very homogeneous distribution of carbon in the alloy. Although Auger Spectroscopy analyses were inconclusive, it is also probable that such powder products have a more homogeneous distribution of impurities (poisons) such as P, Sn, S, and Sb which normally are preferentially segregated at grain boundaries of cast and wrought materials.

A small amount of porosity is apparent in this material, see figures 12(a) and (b). This porosity is probably due to residual argon from the atomization process, rather than incomplete consolidation of the powder particles during extrusion. This conclusion is based on a separate heat treatment study on slugs of the as-extruded powder product which demonstrated that the pore size increased as the solution annealing temperature was increased from 1105° to 1260° C. Such behavior is typical of normal quality, commercially atomized powder products. It is due to argon adsorbed on powder particles and is referred to as Thermally Induced Porosity (TIP). Although these voids are potential trapping sites for hydrogen, the results obtained in this investigation suggest that such voids do not play a major role in determining susceptibility to hydrogen embrittlement, as will be discussed in the CONCLUDING REMARKS section of this report.

Fractographic examinations of specimens tested in air or in hydrogen indicated that fracture modes were similar in both environments, characterized by a ductile, dimple-like structure with full shear lips, see figure 13. Although such dimple-like features are normally characteristic of transgranular fractures, close examination of the size and morphology suggests a primarily intergranular fracture mode along recrystallized grain boundaries.

Udimet 700 condition 5. - This material was solution annealed at 1105° C which is below the 1130° C gamma prime solvus temperature of this material. Consequently, the grain size is slightly smaller (ASTM number 11), see figure 14(a), than that of material condition 4 (ASTM number 10).

The microstructure of the fully heat treated material is shown in figure 14(b) at intermediate magnification and in figure 14(c) at high magnification. As evident from these figures this powder product material does not have either the large intragranular carbides or the continuous network of grain boundary carbide, boride and gamma prime phases that the standard wrought Udimet 700 had. An additional observation can be made from a comparison of the gamma prime morphologies of the powder product in the standard heat treated condition 4 (fig. 12(c)) and in the "yo-yo" heat treated condition 5 (fig. 14(c)). In the latter the gamma prime is more uniformly distributed, slightly smaller in size, and possibly slightly less coherent with the matrix (evidence by a rounded shape) than in the former. Finer (refs. 2 and 10) and less coherent (ref. 11) gamma prime precipitates have been suggested as more effective trapping sites for normally embrittling hydrogen.

Fractographic examinations of specimens tested in air or in hydrogen indicated that fracture modes were similar in both environments, figure 15, and consisted of a ductile, dimple-like structure with complete shear lips. As discussed in connection with the previous powder metallurgy product, this fracture mode is apparently predominately intergranular, as evidenced by the close correlation of the fractographic features with the recrystallized grain size.

CONCLUDING REMARKS

The objective of this investigation, to determine the effect of microstructural features on the susceptibility of Udimet 700 to hydrogen embrittlement, was accomplished. Specifically, a single heat of Udimet 700 was thermomechanically processed to produce five material conditions with identical chemical compositions but with distinct microstructural variations, and then evaluated for resistance to hydrogen embrittlement. All four modified conditions evaluated were more resistant to embrittlement than was the baseline condition, cast plus wrought Udimet 700, under two hydrogen evaluation procedures. However, all material conditions still exhibited some degree of hydrogen embrittlement under the most severe hydrogen evaluation procedure.

Increased resistance to hydrogen embrittlement is attributed to decreased grain size and cleaner grain boundaries. These microstructural features appear to exhibit major beneficial effects. It is also suggested that decreasing gamma prime precipitate size and a lower degree of gamma prime coherency may exert minor beneficial effects. It is probable that these microstructural features, as well as a higher dislocation density in the cold worked material, result in greater trapping of hydrogen because of increased interfacial surface area. Trapped hydrogen is not freely diffusible and thus nonembrittling. Hence, microstructural features which increase internal, interfacial surface area can reduce the amount of hydrogen available for embrittlement (refs. 5, 11, and 12). The effectiveness of these traps is directly related to their respective hydrogen binding energy. The potential role of grain boundary poisons (P, S, Sn, Sb, . . .) could not be determined because of inconclusive Auger Spectroscopy analyses.

These results also suggest that Hydrogen Environment Embrittlement (HEE) and Internal Reversible Hydrogen Embrittlement (IRHE) (refs. 4 and 5) occur by the same mechanism, and differ only with respect to the initial location and supply of hydrogen, and its rate of delivery to and extent of trapping at critical microstructural features. For example, as noted previously, fractographic examinations indicated that specimens exhibited a similar fracture mode whether tested directly in gaseous hydrogen (Hydrogen Environment Embrittlement) or pre-exposed in hydrogen and then tested in air (Internal Reversible Hydrogen Embrittlement).

Furthermore, all five Udimet 700 material conditions were embrittled in the same relative order whether they were tested in hydrogen (HEE) or pre-exposed hydrogen and then tested in air (IRHE). The former hydrogen evaluation procedure did not result in any detectable increase in specimen hydrogen content, while the latter procedure resulted in 20- or 100-fold increases in specimen hydrogen content. In the former case gaseous hydrogen could be supplied continuously to a propagating crack tip, while in the latter case large amounts of hydrogen were distributed homogeneously throughout the specimen microstructure but only a limited amount of readily diffusible hydrogen near stress concentrations could participate in the embrittling process.

Hydrogen Reaction Embrittlement (ref. 5), the formation of new phases within the microstructure, such as methane or molecular hydrogen, was considered unlikely because no microstructural evidence of such new phases was observed for any of the material or evaluation procedures.

As mentioned in the INTRODUCTION the three hydrogenation procedures evaluated in this investigation were selected to span a spectrum of potential service conditions. These conditions ranged from a very short-time exposure with a presumably high surface concentration but a sharp hydrogen gradient inside the specimen and no detectable bulk increase in hydrogen content, to long-time exposures that resulted in homogeneous distributions of hydrogen within specimens to concentration levels expected from extended service in hydrogen environments at lower pressures, temperatures or purity levels.

As also mentioned in the INTRODUCTION one of the self-imposed constraints in this investigation was the evaluation of only practical material conditions appropriate for real structural applications, as opposed to "model" system evaluations. In this regard the heat treatments and the cold working process studied are quite conventional, and the prealloyed powder product is representative of a recent, but practical, advancement in material processing. More importantly the resultant mechanical properties of these material conditions in air are certainly appropriate for many structural applications. For example, both of the powder products (material conditions 4 and 5) exhibited greater notch and smooth tensile strength and ductility than did the baseline condition 1 Udimet 700 alloy. While the ductility of material condition 3 and the strength of condition 2 are slightly below those levels of the baseline Udimet 700, these properties are certainly adequate for some structural applications. Obviously, other mechanical properties such as fatigue, creep strength, rupture life and ductility should be evaluated, where appropriate for intended service uses. This is particularly important for high temperature applications where it is probable that the extremely fine grain size of the powder products will limit use temperature capability to about 700° to 800° C.

It is important to note that both powder product conditions and the cold worked condition of Udimet 700 had significantly greater strength levels than did the baseline, wrought condition, yet they were more resistant to hydrogen embrittlement. These results demonstrate that a commonly believed "rule of thumb," that higher strength materials are more susceptible to embrittlement than are lower strength materials, is not always valid (ref. 4). In fact there are now many investigations (refs. 3 to 5, 11 and 12) demonstrating that low strength materials can be severely embrittled, particularly in hydrogen gas, that continued acceptance of such a "rule of thumb" could result in catastrophic service failures.

Microstructural variables have been shown to play dramatic roles in determining the susceptibility of Udimet 700 to hydrogen embrittlement. Cleaning up grain boundaries and decreasing grain size have major beneficial effects, while intragranular gamma prime size and morphology appear to have minor beneficial effects. By consideration of these microstructural features, it is suggested that nickel-base superalloys with specific microstructures may provide reliable service for some types of structural applications in hydrogen environments.

SUMMARY OF RESULTS

The objective of this investigation was to alter the grain size and distribution of grain boundary phases and impurities by various thermomechanical processing steps in order to minimize the susceptibility of Udimet 700 to hydrogen embrittlement. Material from a single heat of wrought Udimet 700 was processed and/or heat treated to produce five material conditions with identical compositions but with distinct microstructural variations. The alloy was evaluated in three wrought forms - standard heat treatment, modified heat treatment, and standard heat treatment plus an intermediate cold working step. The alloy was also evaluated in two extruded from atomized powder forms - standard heat treatment, and a "yo-yo" heat treatment.

These five material conditions were then subjected to baseline smooth and notched tensile tests in air or argon at 23° and 675° C and to three hydrogenation procedures. Relative susceptibility of these five material conditions to embrittlement by hydrogen was correlated with various microstructural features by appropriate metallographic techniques. The following results were obtained:

1. Both of the Udimet 700 powder products exhibited excellent resistance to embrittlement under the mild and intermediate severity hydrogenation procedures. No significant degradation was observed in notch or smooth tensile strengths, and ductilities averaged about 25 percent reduction of area. The ductility of these materials were reduced to about 15 percent reduction of the area when tested under the most severe hydrogen evaluation procedure.

2. The high tolerance for hydrogen exhibited by these powder product materials is attributed to their fine grain sizes (ASTM 10 and 11) and clean, almost precipitate-free grain boundaries. In addition the baseline tensile strengths and ductility levels of these powder products in air were superior to the properties of the standard wrought Udimet 700.

3. The powder product material given the "yo-yo" heat treatment, which involved solution annealing below the material's gamma prime solvus, had slightly higher resistance to hydrogen embrittlement than did the material given the standard heat treatment. This increased resistance to embrittlement is attributed to a finer grain size (ASTM 11) and slightly finer, more uniform sized, and possibly less coherent intragranular gamma prime precipitates.

4. The resistance to hydrogen embrittlement of wrought Udimet 700 could be substantially improved by cold working which resulted in deformed grains and gamma prime precipitates, and enhanced carbide precipitation on intragranular deformation bands and dislocation networks. This material condition was stronger but less ductile in air than the baseline standard Udimet 700 material.

5. The resistance of wrought Udimet 700 was also improved by eliminating the carbide precipitation heat treating step from the standard heat treating schedule, thereby reducing the amounts of grain boundary phases. This material condition had a lower strength level but higher ductility in air than did the standard Udimet 700.

6. Hydrogen analyses of fractured specimens indicated that, for a given hydrogenation procedure, all five material conditions absorbed identical amounts of hydrogen. Hence, relative susceptibility to embrittlement cannot be attributed to varying absorption rates. Tensile testing directly in hydrogen did not result in any detectable increase in hydrogen content, while the two hydrogen exposures at 675° C resulted in 20- and 100-fold increases (to 12 and 60 ppm), respectively.

7. The results of this study support the contention that Hydrogen Environment Embrittlement and Internal Reversible Hydrogen Embrittlement occur by the same mechanism, and differ only with respect to the initial location and supply of hydrogen, and its rate of delivery to and extent of trapping at critical microstructural features.

REFERENCES

1. Frick, V., Janser, G. R., and Brown, J. A., "Enhanced Flaw Growth in SSE Main Engine Alloys in High Pressure Gaseous Hydrogen," Space Shuttle Materials, Society of Aerospace Materials and Process Engineers, Azusa, Calif., 1971, pp. 597-634.

2. Gray, H. R., and Joyce, J. P., "Hydrogen Environment Embrittlement of Turbine Disk Alloys," in *Effect of Hydrogen on Behavior of Materials*, A. W. Thompson and I. M. Bernstein, eds., AIME, 1976, pp. 578-588. (also NASA TN D-8046, 1975).
3. Harris, J. A., Jr. and Van Wanderham, M. C., "Properties of Materials in High Pressure Hydrogen at Cryogenic, Room and Elevated Temperatures," Pratt & Whitney Research and Development Center, FR-5768, NASA CR-124394, 1973.
4. Jewett, R. P., Walter, R. J., Chandler, W. T., and Frohmberg, R. P., "Hydrogen Environment Embrittlement of Metals," NASA CR-2163, 1973.
5. Gray, H. R., "Testing for Hydrogen Environment Embrittlement: Experimental Variables," *Hydrogen Embrittlement Testing*, STP-543, American Society for Testing and Materials, 1974, pp. 133-151.
6. *Aerospace Structural Metals Handbook*, Vol. 5 - Non-Ferrous Heat Resistant Alloys, AFML-TR-68-115, Belfour Stulen, Inc., 1977, pp. 4207-1 to 4207-8.
7. Athey, R. L., and Moore, J. B., "Development of Astroloy Disks for Advanced Jet Engine Application," Pratt & Whitney Aircraft, PWA-GP-67-21, 1967.
8. Gordon, W. A., and Chapman, G. B., "Quantitative Direct-Current Arc Analysis of Random Compositions of Microgram Residues in Silver Chloride Common Matrix," NASA TN D-5532, 1969.
9. Gray, H. R., "Ion and Laser Microprobes Applied to the Measurement of Corrosion Produced Hydrogen on a Microscopic Scale," *Corrosion*, Vol. 28, No. 2, Feb. 1972, pp. 47-54.
10. Gray, H. R., "Embrittlement of Nickel-, Cobalt-, and Iron-Base Superalloys by Exposure in Hydrogen," NASA TN D-7805, 1975.
11. Thompson, A. W., and Brooks, J. A., "Hydrogen Performance of Precipitation - Strengthened Stainless Steels Based on A-286," *Metallurgical Transactions A*, Vol. 6A, July 1975, pp. 1431-1442.
12. Bernstein, I. M., and Thompson, A. W., "Resisting Hydrogen Embrittlement," *Alloy and Microstructural Design*, J. K. Tien, and G. S. Ansell, eds., Academic Press, 1976, Chapter 11, pp. 303-347.

**REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR**

TABLE I. - UDIMET 700 CONDITIONS, HEAT TREATMENTS, AND MICROSTRUCTURAL FEATURES

Material condition	Heat treatment		Average grain size		Grain boundary carbides and γ'	Intragranular* γ' size, μm
	Temperature, $^{\circ}\text{C}$	Time, hr	ASTM No.	Equiv. diam, μm		
1 1.9-cm-diam wrought bar (standard heat treatment)	1175 1080 845 760	4 4 24 16	2	180	Large, numerous and almost continuous	0.08 and 0.35
2 1.9-cm-diam wrought bar (modified heat treatment)	1175 1080 760	4 4 16	2	180	Large but scattered	0.08 and 0.35
3 Wrought bar swaged 50 percent to 1.3-cm-diam bar (standard heat treatment)	1175 1080 Cold working 845 760	4 4 24 16	~3	125	Large, numerous and almost continuous	0.08 and 0.35
4 Atomized powder, extruded to 1.1-cm-diam bar (standard heat treatment)	1175 1080 845 760	4 4 24 16	10	11	Small, few and scattered	0.08 and 0.35
5 Atomized powder, extruded to 1.1-cm-diam bar (yo-yo heat treatment)	1105 870 960 650 760	4 8 4 24 8	11	8	Small, few and scattered	0.04 and 0.3

* Total ~35 v/o γ' , with varying proportions of each size.

TABLE II. - UDIMET 700 COMPOSITIONS

Material conditions	Source of analyses	Weight percent											ppm (by weight)		
		Ni	Fe	Cr	Co	Al	Ti	Mo	B	Si	Mn	C	N	O	H
Ladle analysis (wrought bar stock)	Nominal (ref. 6) range	Bal.	0.4	14-16	17-20	3.75-4.75	2.75-3.75	4.5-6.0	0.025-0.035	----	----	0.03-0.10	--	--	---
	Vendor certification	Bal.	0.19	14.6	19.0	4.46	3.38	4.85	0.027	<0.1	<0.1	0.07	--	--	---
Atomized powder	Vendor certification	----	----	----	----	----	----	----	----	----	----	----	29	90	---
Wrought (1, 2, and 3)	This investigation	Bal.	0.14	13.9	18.9	5.9	3.2	6.1	<0.06	0.01	<0.001	0.061	28	7	0.6
Extruded powder (4 and 5)	This investigation	Bal.	0.14	14.5	19.0	6.1	3.1	5.9	<0.06	0.01	<0.001	0.061	46	83	0.6

TABLE III. - EVALUATION PROCEDURES

Evaluation procedure	Exposure at 675° C/100 hr	Tensile test atmosphere(s) and temperature(s)
A	None	3.5 MN/m ² hydrogen, 23° and 675° C Baseline in ambient air, 23° and 675° C
B	0.1 MN/m ² hydrogen Baseline in argon	} Ambient air, 23° and 675° C
C	3.5 MN/m ² hydrogen with creep stress, 690 MN/m ² Baseline in air with creep stress, 690 MN/m ²	

TABLE IV. - TEST RESULTS FOR SPECIMENS TENSILE TESTED IN HYDROGEN OR AIR

(EVALUATION PROCEDURE A)

Specimen		Tensile test conditions			Tensile strength		Elongation, percent	Reduction of area, percent	Hydrogen content, ppm		
Number	Type	Gas	Pressure, MN/m ²	Temperature, °C	MN/m ²	ksi					
UDIMET 700 - STANDARD HEAT TREATMENT (MATERIAL CONDITION 1)											
24	Notched ↓	Air	0.1	23 ↓	1595	231	---	---	0.5, 0.5, 0.6		
25						1620	235	---		---	
34						1615	234	---		---	
36						1580	229	---		---	
26		H ₂	3.5			1060	154	---		---	
27						1115	162	---		---	
77						1165	169	---		---	
28						1370	199	---		---	
33		Air	.1			675	1515	220		---	---
35		Air	.1			675	1540	223		---	---
22		H ₂	3.5	675	1475	214	---	---			
10	Smooth ↓	Air	.1	23	1400	203	22	25	0.4		
11		Air	.1	23	1400	203	22	23			
3		H ₂	3.5	23	1055	153	4	7			
14		Air	.1	675	1205	175	25	31			
15		Air	.1	675	1185	172	22	33			
4			H ₂	3.5	675	1205	175	19		22	
UDIMET 700 - MODIFIED HEAT TREATMENT (MATERIAL CONDITION 2)											
80	Notched ↓	Air	0.1	23 ↓	1400	203	---	---	---		
127		Air	.1			1455	211	---		---	
79		H ₂	3.5			1440	209	---		---	
125		H ₂	3.5			1395	202	---		---	
128	↓	Air	.1	675 ↓	1365	198	---	---	---		
126		H ₂	3.5			1315	191	---		---	
119		Air	.1		23	1345	195	29		29	
115		H ₂	3.5		23	1305	189	16		18	
120	↓	Air	.1	675 ↓	1185	172	22	25	---		
116		H ₂	3.5		675	1215	176	16		15	

REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR

TABLE IV. - Concluded.

Specimen		Tensile test conditions			Tensile strength		Elongation, percent	Reduction of area, percent	Hydrogen content, ppm
Number	Type	Gas	Pressure, MN/m ²	Temperature, °C	MN/m ²	ksi			
UDIMET 700 - COLD WORKED AND STANDARD HEAT TREATMENT (MATERIAL CONDITION 3)									
48	Notched	Air	0.1	23	2105	305	---	---	0.2
113	↓	Air	.1	↓	2110	306	---	---	
49		H ₂	3.5		1985	288	---	---	
106		H ₂	3.5		2095	304	---	---	
114		H ₂	3.5		2035	295	---	---	
47		Air	.1	675	1960	284	---	---	
50	↓	H ₂	3.5	675	1995	289	---	---	
51		H ₂	3.5	675	1970	286	---	---	
40		Air	.1	23	1870	271	6	12	
109		Air	.1	23	1780	258	10	15	
41		H ₂	3.5	23	1830	265	6	9	
39	↓	Air	.1	675	1565	227	11	22	
101		H ₂	3.5	675	1570	228	6	14	
44		H ₂	3.5	675	1620	235	3	9	
UDIMET 700 - EXTRUDED POWDER-STANDARD HEAT TREATMENT (MATERIAL CONDITION 4)									
62	Notched	Air	0.1	23	1615	233	---	---	0.6
69	↓	H ₂	3.5	23	1620	235	---	---	
90		H ₂	3.5	23	1675	243	---	---	
68		Air	.1	675	1625	236	---	---	
70		H ₂	3.5	675	1595	231	---	---	
63	↓	Air	.1	23	1455	211	28	37	
85		Air	.1	23	1525	221	26	31	
64		H ₂	3.5	23	1490	216	21	20	
82		Air	.1	675	1235	179	26	31	
81		Air	.1	675	1250	181	19	26	
66	↓	H ₂	3.5	675	1450	210	24	25	
UDIMET 700 - EXTRUDED POWDER-YOYO HEAT TREATMENT (MATERIAL CONDITION 5)									
56	Notched	Air	0.1	23	1730	255	---	---	0.8
53	↓	H ₂	3.5	23	1795	260	---	---	
100		H ₂	3.5	23	1725	249	---	---	
55		Air	.1	675	1635	237	---	---	
54		H ₂	3.5	675	1635	237	---	---	
91	↓	Air	.1	23	1505	218	30	36	0.5
58		H ₂	3.5	23	1580	229	18	26	
92		Air	.1	675	1255	182	21	24	
59		H ₂	3.5	675	1325	192	22	26	

TABLE V. - TEST RESULTS FOR SPECIMENS EXPOSED IN HYDROGEN OR ARGON, FOLLOWED BY TENSILE TESTING IN AIR (EVALUATION PROCEDURE B)

Specimen		Exposure conditions at 675° C			Post-exposure tensile test in air					Hydrogen content, ppm	
Number	Type	Gas	Pressure, MN/m ²	Time, hr	Temperature, °C	Strength		Elongation, percent	Reduction of area, percent		
						MN/m ²	ksi				
UDMET 700 - STANDARD HEAT TREATMENT (MATERIAL CONDITION 1)											
21	Notched ↓	Argon	0.1 ↓	100 ↓	23	1525	221	--	--	{ 0.5, 0.5 0.8, 0.8 12, 12	
30		Argon			23	1640	238	--	--		
31		H ₂			23	1615	234	--	--		
23		Argon			675	1505	218	--	--		
32		H ₂			675	1525	221	--	--		
16	Smooth ↓	Argon	↓	102 ↓	23	1385	201	20	22		
20		H ₂			23	1400	203	16	16		
6		Argon			675	1240	180	22	23		
17		H ₂			675	1240	180	21	23		
UDMET 700 - MODIFIED HEAT TREATMENT (MATERIAL CONDITION 2)											
129	Notched ↓	Argon	0.1 ↓	100 ↓	23	1505	218	--	--		
130		H ₂			23	1475	214	--	--		
131		Argon			675	1360	197	--	--		
132		H ₂			675	1400	203	--	--		
118		Argon			23	1330	193	26	25		
122	Smooth ↓	H ₂	↓	102 ↓	23	1305	189	19	19		
123		Argon			675	1205	175	25	24		
124		H ₂			675	1195	173	26	24		
UDMET 700 - COLD WORKED AND STANDARD HEAT TREATMENT (MATERIAL CONDITION 3)											
104	Notched ↓ Smooth ↓	Argon	0.1 ↓	100 ↓	23	2095	304	--	--		12, 12
111		Argon			↓	2100	305	--	--		
112		H ₂			↓	1925	279	--	--		
52		H ₂			↓	1850	268	--	--		
105		Argon			675	1925	279	--	--		
45		H ₂			675	1880	273	--	--		
103		Argon			23	1870	271	6	8		
107		Argon			↓	1815	263	9	10		
108		H ₂			102	1795	260	6	8		
37		H ₂			102	1850	268	5	7		
		Argon			100	675	1565	227	11	22	
38		H ₂			102	675	1565	227	14	24	

* Test not performed—data estimated from table IV.

REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR

TABLE V. - Concluded.

Specimen		Exposure conditions at 675° C			Post-exposure tensile test in air					Hydrogen content, ppm
Number	Type	Gas	Pressure, MN/m ²	Time, hr	Temperature, °C	Strength		Elongation, percent	Reduction of area, percent	
						MN/m ²	ksi			
UDIMET 700 - EXTRUDED POWDER-STANDARD HEAT TREATMENT (MATERIAL CONDITION 4)										
88	Notched ↓ Smooth	Argon	0.1 ↓	100	23	1780	258	--	--	12, 12
89		H ₂		101	23	1760	255	--	--	
86		Argon		100	675	1495	217	--	--	
87		H ₂		101	675	1545	224	--	--	
83	Smooth	Argon	100	23	1515	220	24	35		
65	Smooth	H ₂	101	23	1460	212	16	24		
UDIMET 700 - EXTRUDED POWDER-YOYO HEAT TREATMENT (MATERIAL CONDITION 5)										
96	Notched ↓ Smooth	Argon	0.1 ↓	100	23	1780	258	--	--	11, 11
97		H ₂		101	23	1780	258	--	--	
98		Argon		100	675	1545	224	--	--	
99		H ₂		101	675	1550	225	--	--	
94	Smooth	Argon	100	23	1580	229	26	29		
57	Smooth	H ₂	101	23	1545	224	22	28		

1-9574

TABLE VI. - TEST RESULTS FOR SMOOTH SPECIMENS EXPOSED IN HYDROGEN OR AIR AT A CREEP STRESS OF 690 MN/m^2 , FOLLOWED BY TENSILE TESTING IN AIR AT 23°C (EVALUATION PROCEDURE C)

Specimen number	Exposure conditions				Post-exposure tensile test				Hydrogen content, ppm
	Gas	Pressure, MN/m ²	Time, hr	Creep strain, percent	Strength		Elongation, percent	Reduction of area, percent	
					MN/m ²	ksi			
UDIMET 700 - STANDARD HEAT TREATMENT (MATERIAL CONDITION 1)									
5	Air	0.1	100	<1	1400	203	18	20	59
1	H ₂	3.5	137	1.3	1260	183	8	9	
2	H ₂	3.5	160	.2	1270	184	8	9	
UDIMET 700 - MODIFIED HEAT TREATMENT (MATERIAL CONDITION 2)									
121	Air	0.1	101	<1	1380	200	26	24	
117	H ₂	3.5	101	.2	1305	189	15	15	
UDIMET 700 - COLD WORKED AND STANDARD HEAT TREATMENT (MATERIAL CONDITION 3)									
102	Air	0.1	100	<1	1860	270	7	11	60
43	H ₂	3.5	141	.4	1830	265	2	6	
UDIMET 700 - EXTRUDED POWDER-STANDARD HEAT TREATMENT (MATERIAL CONDITION 4)									
84	Air	0.1	100	<1	1550	225	24	32	63
67	H ₂	3.5	115	0.2	1455	211	11	15	
UDIMET 700 - EXTRUDED POWDER-YOYO HEAT TREATMENT (MATERIAL CONDITION 5)									
93	Air	0.1	100	<1	1565	227	26	25	66
61	H ₂	3.5	114	.1	1560	226	12	14	

TABLE VII. - SUMMARY OF HYDROGEN EFFECTS DETERMINED IN THIS INVESTIGATION

[x = >3 percent reduction in tensile strength; y = >10 percent decrease in reduction of area; - = insignificant change in strength/ductility; ND = not determined-insufficient quantity of material.]

Material condition	Evaluation procedure A (tensile test in hydrogen)				Evaluation procedure B (H ₂ , exposure 675 ^o C/100 hr)				Evaluation procedure C (H ₂ creep exposure, 675 ^o C, 690 MN/m ² stress)
	Notched		Smooth		Notched		Smooth		Smooth at 23 ^o C
	23 ^o C	675 ^o C	23 ^o C	675 ^o C	23 ^o C	675 ^o C	23 ^o C	675 ^o C	
Std. H. T. (1)	x	-	xy	-y	-	-	-y	--	xy
Mod. H. T. (2)	-	-	-y	-y	-	-	-y	--	xy
Cold work (3)	x	-	-y	-y	x	-	-y	--	-y
Powder + Std. H. T. (4)	-	-	-y	--	-	-	-y	ND	xy
Powder + YoYo H. T. (5)	-	-	-y	--	-	-	--	ND	-y

REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR

REPRODUCIBILITY OF THE ORIGINAL DATA IS POOR

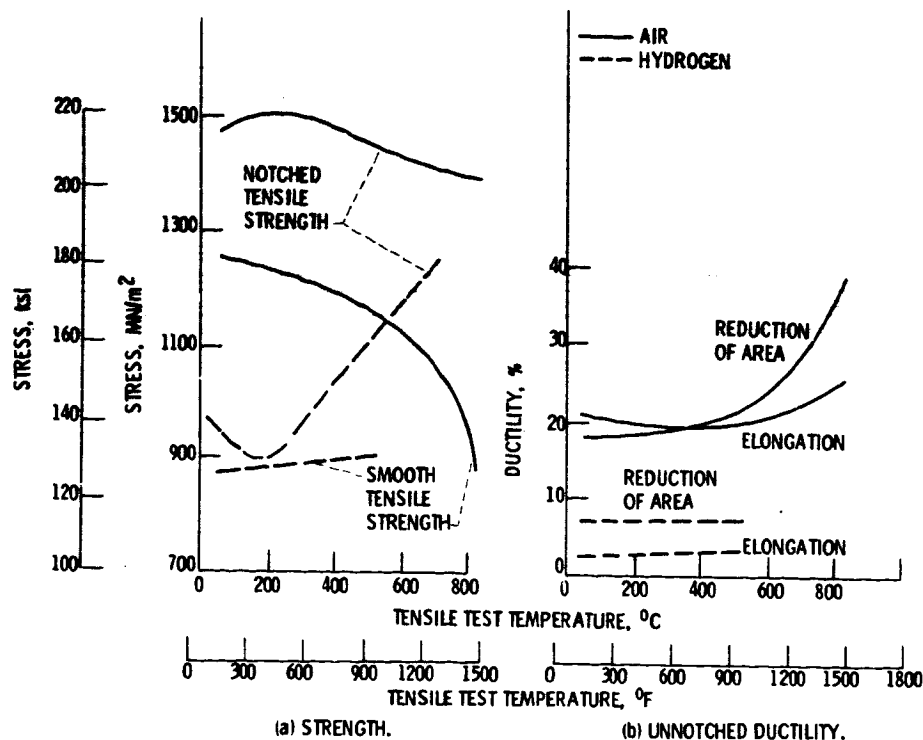


Figure 1. - Effect of test temperature on tensile properties of Udimet 700 at hydrogen pressure of 31 to 52 MN/m² (4500 to 7500 psi). Notched specimens; stress concentration factor $K_t = 8$ (ref. 1).

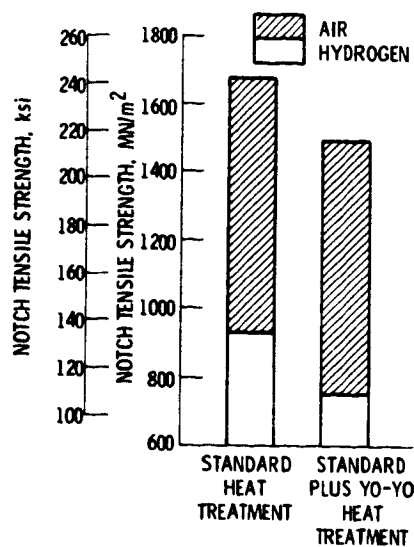


Figure 2. - Notch tensile strength of wrought Udimet 700 in 3.5 MN/m² hydrogen or ambient air at 23°C; stress concentration factor $K_t \geq 6$ (ref. 2).

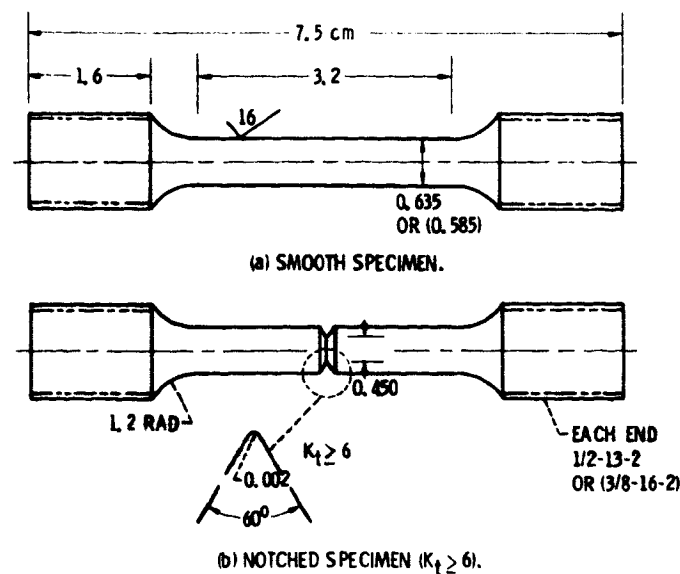


Figure 3. - Test specimens used in this investigation. (All dimensions except threads in cm.)

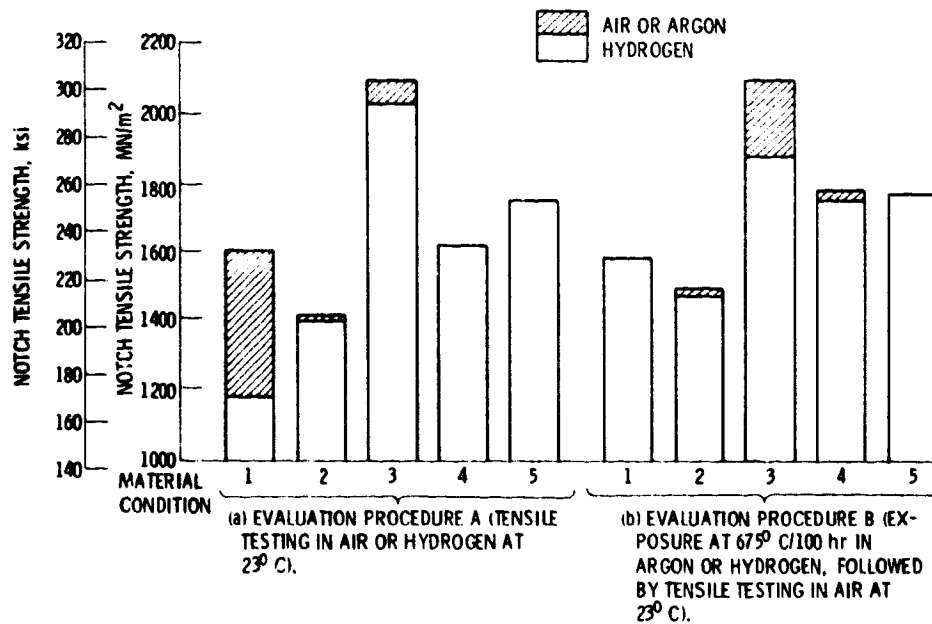


Figure 4. - Effect of hydrogen evaluation procedures on room temperature notch tensile strength of Udimet 700 material conditions.

REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR

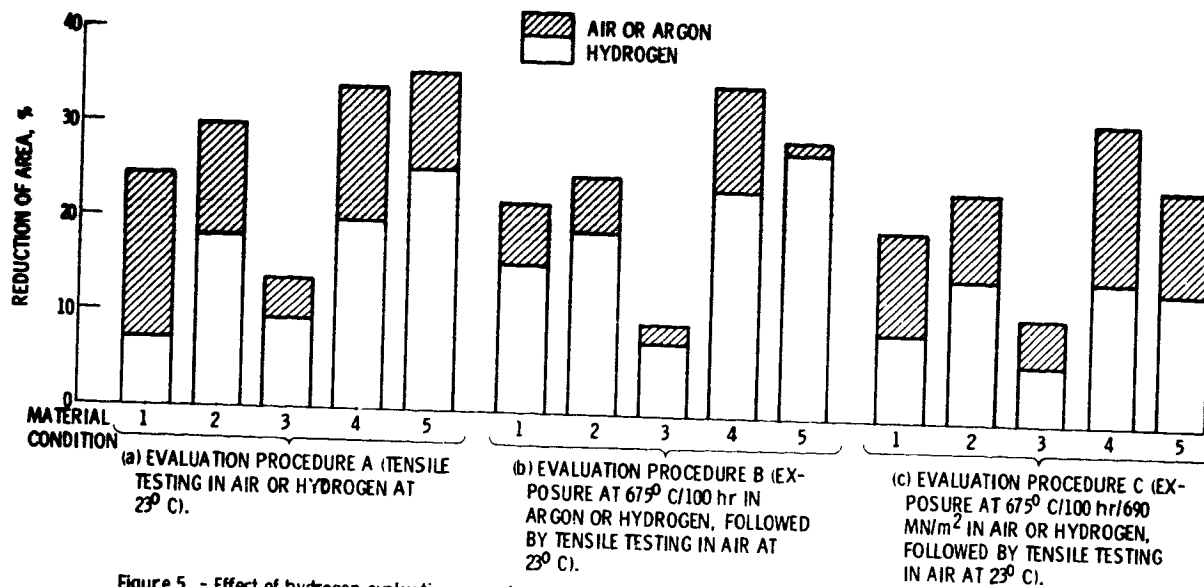


Figure 5. - Effect of hydrogen evaluation procedures on room temperature tensile ductility of Udimet 700 material conditions.

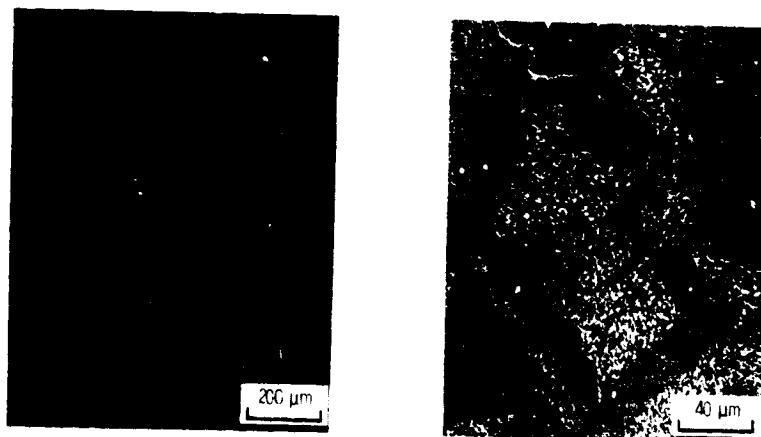


Figure 6. - Microstructure of as-received Udimet 700 alloy (material condition 1).

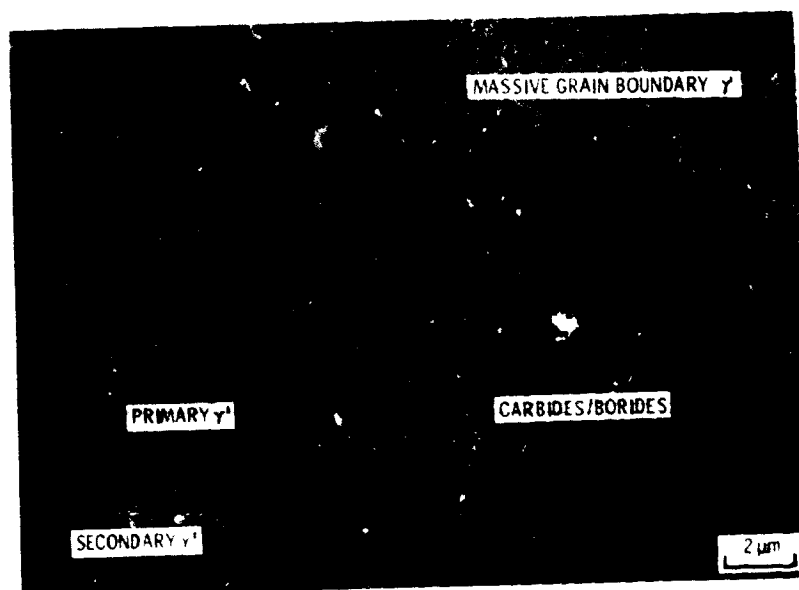
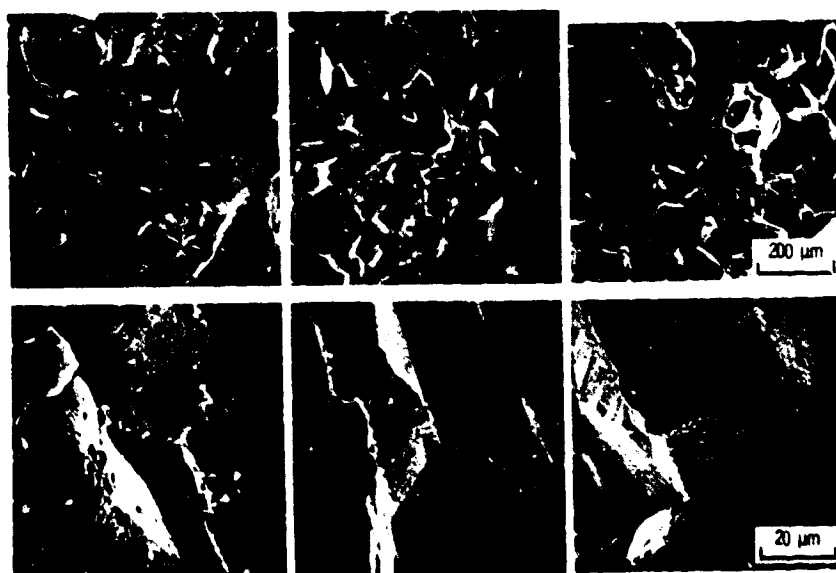


Figure 6. - Concluded.



(a) TESTED IN AIR, 25 PERCENT RA (EVALUATION PROCEDURE A). (b) TESTED IN HYDROGEN, 7 PERCENT RA (EVALUATION PROCEDURE A). (c) EXPOSED IN HYDROGEN, 9 PERCENT RA (EVALUATION PROCEDURE C).

Figure 7. - Scanning electron fractographs of Udimet 700 (material condition 1).

REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR

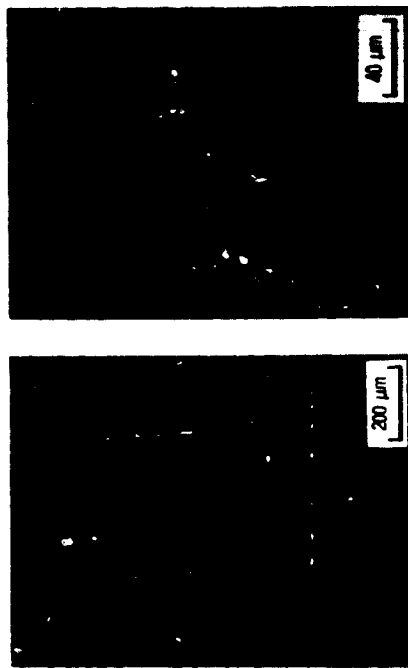


Figure 8. - Microstructure of Udimet 700 alloy heat-treated without 845° C carbide precipitation step (material condition 2).

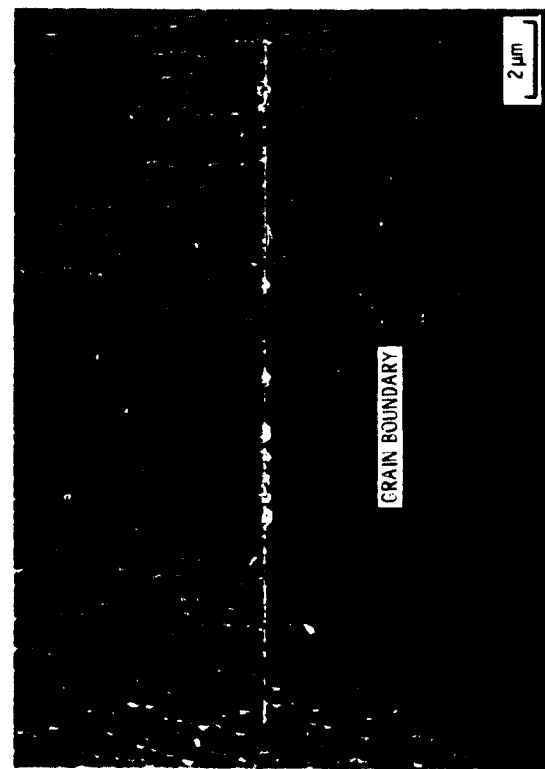


Figure 8. - Concluded.

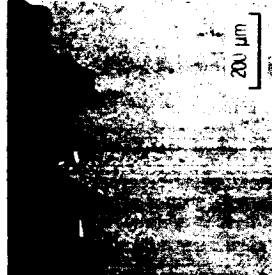
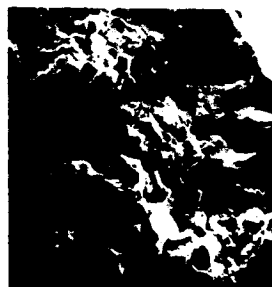


Figure 9. - Scanning electron fractographs and optical micrograph of Udimet 700 (material condition 2), tested in hydrogen, 18 percent RA (evaluation procedure A).

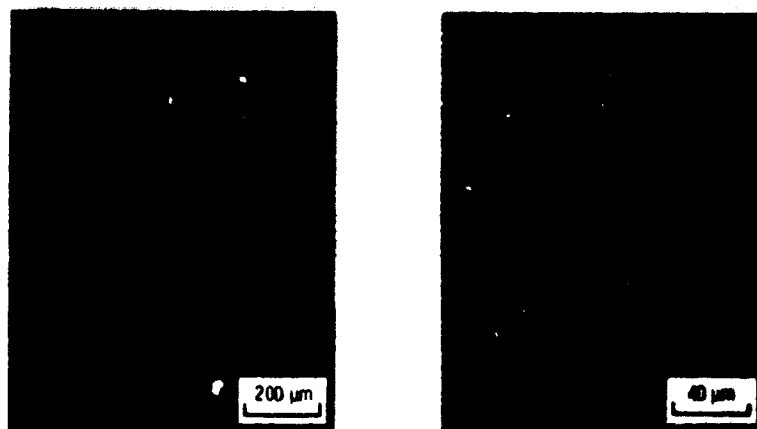


Figure 10. - Microstructure of as-received Udimet 700 alloy with 50 percent cold work (material condition 3).

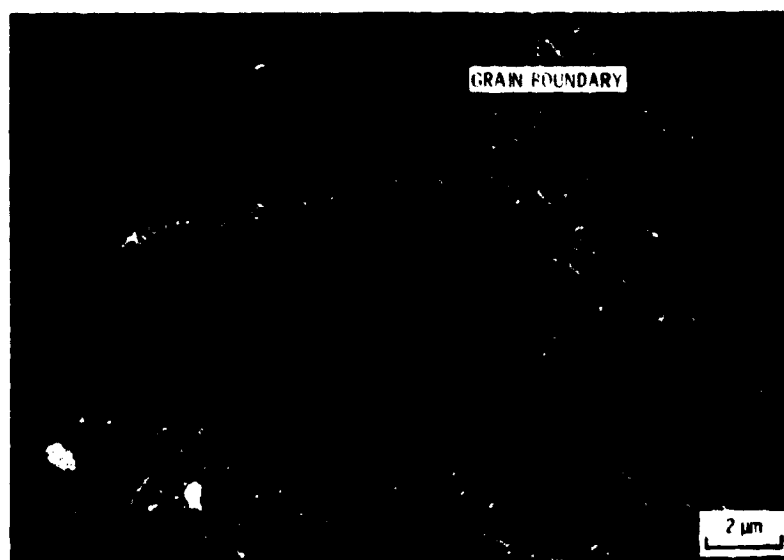
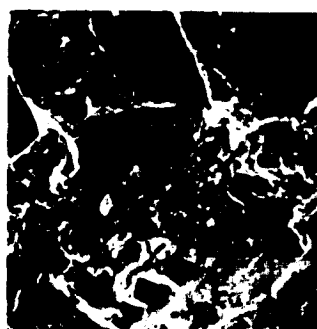
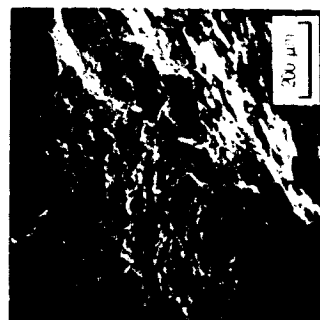


Figure 10. - Concluded.

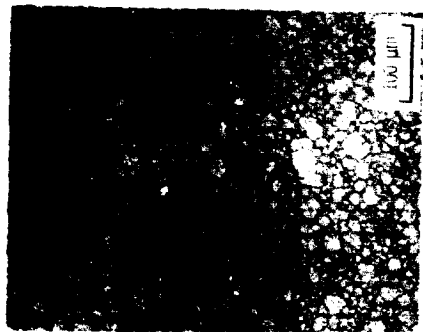


(a) TESTED IN AIR, 12 PERCENT RA (EVALUATION PROCEDURE A).

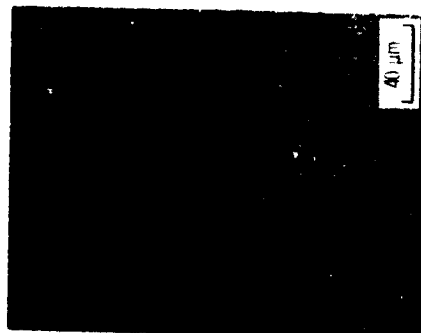


(b) TESTED IN HYDROGEN, 9 PERCENT RA (EVALUATION PROCEDURE A).

Figure 11. - scanning electron fractographs of Udimet 700 (material condition 3).



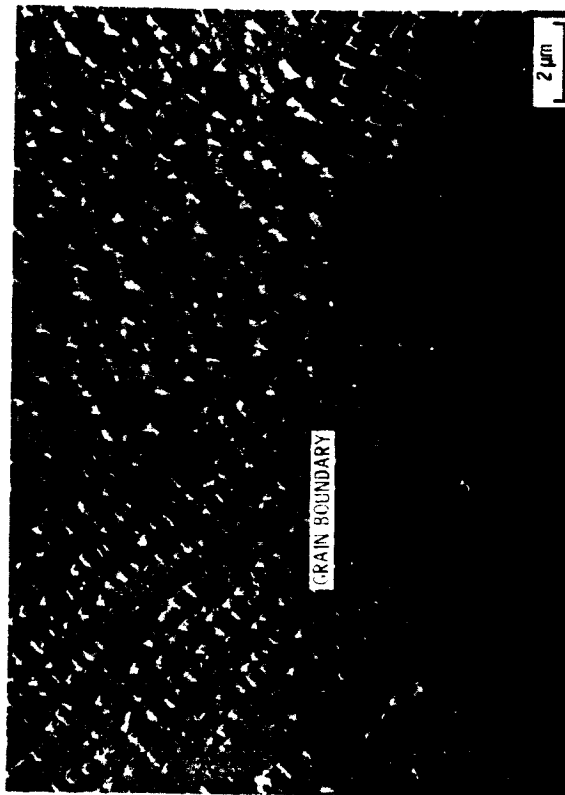
(a) SOLUTION ANNEALED AT 1175° C.



(b) FULLY HEAT TREATED.

Figure 12. - Microstructure of powder metallurgy Udimet 700 after solution annealing and after standard heat treatment (material condition 4).

REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR



(c) FULLY HEAT TREATED.

Figure 12. - Concluded.



(a) TESTED IN AIR, 37 PERCENT RA (EVALUATION PROCEDURE A).



(b) TESTED IN HYDROGEN, 20 PERCENT RA (EVALUATION PROCEDURE A).

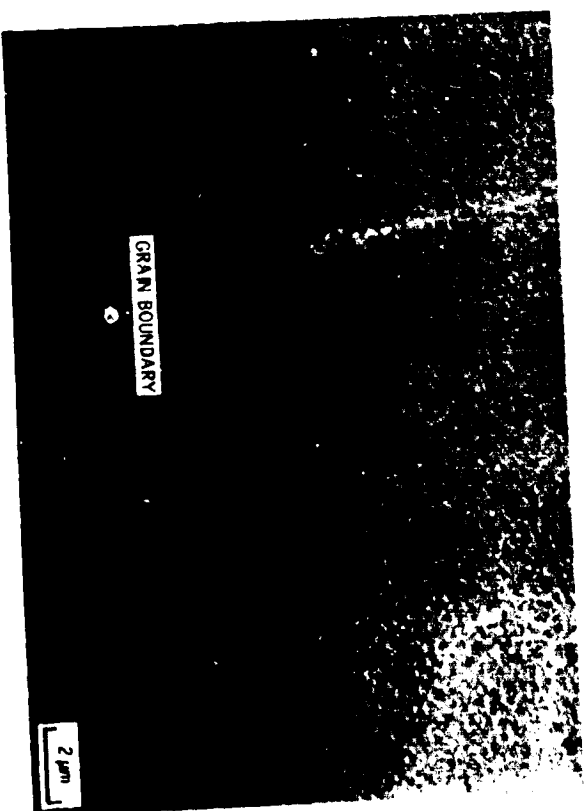
Figure 13. - Scanning electron micrographs of Udimet 700 (material condition 4i).



(a) SOLUTION TREATED 1100°C.



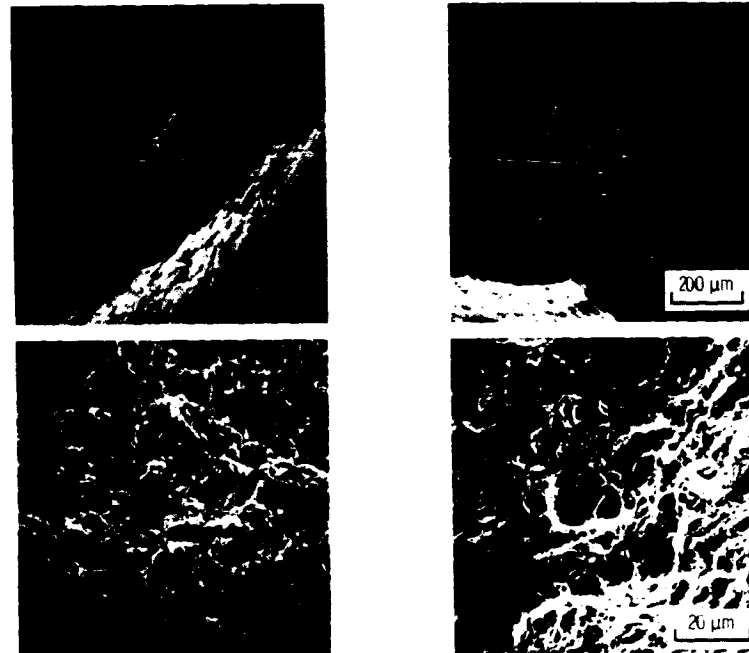
Fig. 14. - Surface texture of powder metallurgy Udimet 700 after solution treatment and air cooling. (material condition 5i).



(c) HEAT TREATED.

Figure 14. - Continued.

REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR



(a) TESTED IN AIR, 36 PERCENT RA
(EVALUATION PROCEDURE A).

(b) TESTED IN HYDROGEN, 26 PERCENT
RA (EVALUATION PROCEDURE A).

Figure 15. - Scanning electron fractographs of Udimet 700 (material condition 5).

END

DATE

FILMED

JUN

22 1978